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Characterization of Nanostructured Polymer Films

RODNEY PRIESTLEY
TRUSTEES OF PRINCETON UNIVERSITY

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Final Report

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14. ABSTRACT

In 2011, we discovered that polymer films with exceptional thermal and kinetic stability could be formed by Matrix Assisted Pulsed Laser Evaporation (MAPLE) onto substrates held at low temperatures compared to the glass transition temperature (Tg) of the polymer. The unique and unprecedented combination of properties is due to the film morphology, i.e., the films are nanostructured. The aim of this proposal was to understand the mechanism of film formation and to characterize the nanoscale building blocks of the stable glasses. Recently, we characterized the transport, i.e., time-of-flight, and nanoscale thermal properties of amorphous polymer nanoglobules fabricated via Matrix-Assisted Pulsed Laser Deposition (MAPLE). We discovered that stability in nanostrutured glasses was due to the nanoscale stability of the materials' building blocks, i.e., nanoglobules.

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AFOSR YIP: Characterization of Nanostructured Polymer Films

Rodney D. Priestley, Chemical and Biological Engineering, Princeton University, Princeton, NJ 08544

Final Report

I. Summary

In 2011, we discovered that polymer films with exceptional thermal and kinetic stability could be formed by Matrix Assisted Pulsed Laser Evaporation (MAPLE) onto substrates held at low temperatures compared to the glass transition temperature (T_g) of the polymer. The unique and unprecedented combination of properties is due to the film morphology, *i.e.*, the films are nanostructured, as illustrated in Figure 1. We have spent the past few years trying to understand the growth mechanism of film formation and directly connecting the nanostructure to overall film properties. This final report focuses on the mechanism of nanostructured stable glass formation and the properties of the nanoscale building blocks. We will also discuss our recent results on using MAPLE to engineer the crystal structure in thin films.

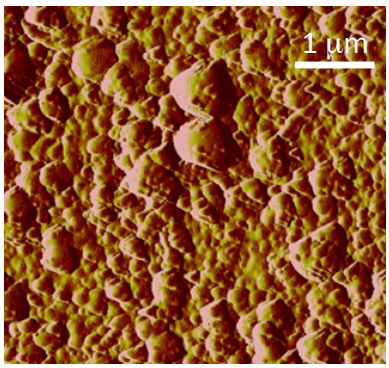

Figure 1. Surface topology of MAPLE-deposited PMMA film reveals the presence of surface nanoglobules.

Energy Landscape of Glass. The development of Matrix Assisted Pulsed Laser Evaporation (MAPLE) has enabled the gentle deposition of large macromolecules, where degradation can be avoided during laser processing. In the MAPLE method, a pulsed laser ablates a frozen target solution under vacuum, forming a plume, which is collected on a temperature-controlled substrate. As illustrated in Figure 1, we are able to generate, what we are calling, structured glasses. These glasses produced by MAPLE are significantly different from those formed by supercooling, *i.e.*, the normal route to the glassy state. The differences in MAPLE-deposited glasses and those produced by supercooling are illustrated in Figure 2.

Surface Stability. We have suggested that the enhanced thermal stability of the nanostructured PMMA films is a direct result of the properties of the nanoglobular building blocks of film formation, i.e., the nanoglobules are thermally stable. We have shown that complete coalescence of surface nanoglobules occurs only at temperatures well above the normal T_g. In addition, we have indirectly investigated the thermal stability of the nanoglobules by examining the surface morphology via AFM of a series of PMMA films deposited at various substrate temperatures (see Figure 3). We note that the substrate temperature was the only processing parameter varied during these experiments, and AFM experiments were conducted at ambient conditions. The root-mean-square (RMS) roughness was observed to decrease from ~ 140 nm at a low substrate temperature to ~ 2 nm for a substrate temperature of 393 K, as the surface nanostructure becomes less prominent. While the T_g of the 5 K PMMA was 365 K (as determined by DSC on second heating at heating rate of 10 K/min), we observed that surface nanoglobules were clearly present at 373 K. The fact that surface globules were still present at 373 K is significant. At the T_g, the polymer segmental relaxation time is approximately 100 s. The total deposition time of all experiments was ~ 3600 s. This indicates that sufficient time was allotted during the growth of the film for complete polymer chain relaxation, including relaxation of surface features. The presence of intact surface globules at a substrate temperature of 373 K suggests that the film has a T_g greater than 373 K, i.e., an enhanced T_g compared to the normal value of 365 K. Our results suggest that complete surface coalescence of the nanoglobules does not occur until the substrate temperature is at least 393 K. We note that at 393 K, the segmental relaxation time of the bulk polymer would be ~ 0. 1–1 s.

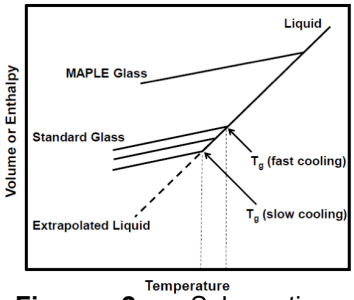

Figure 2. Schematic of volume or enthalpy vs. temperature comparing standard and MAPLE-deposited glasses.

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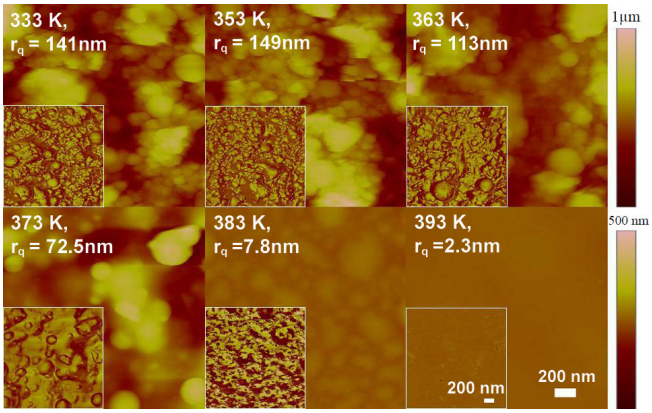

Figure 3. AFM height images and phase images (insets) of PMMA films deposited via MAPLE onto silica substrates held at various temperatures. The substrate temperature and RMS roughness for each film are given in the upper left corner of each panel. Nanoglobules persist on the surface at temperatures well above the bulk T_g of 5K PMMA. A substrate temperature of 393K is required to fully eliminate surface nanoglobules.

Mechanism of MAPLE: Cooling Rate. We have argued that the significant enhancement in T_g of MAPLE-deposited glasses is due to the cooling rate (Q) experienced by the molecules during deposition. To quantitatively estimate the quench rate, we measured the fictive temperature (T_f) as a function of cooling rate for PMMA and used the VFT expression to determine the cooling rate required to achieve the T_f of MAPLE-deposited films. Data for the normalized fictive temperature (calculated by the Moynihan method) versus normalized cooling rate is shown on an Arrhenius plot in Figure 4. Cooling rate and fictive temperature are normalized by a standard cooling rate of 20 K/min, and the respective T_f obtained at that cooling rate for each polymer. On the second set of axes in Figure 4, an Arrhenius plot of the VFT equation is superimposed. In the normalized form, the fictive temperatures of MAPLE-deposited glasses range from $T_{f,s}/T_f$ = 0.8 - 0.9. These values correspond approximately to $\log(Q/Q_s)$ = 7–10, implying an effective cooling rate between 10^8 –10^{11} K/min. The shaded region in Figure 4 indicates the range of $T_{f,s}/T_f$ values observed for MAPLE-deposited PMMA. Such high cooling rates would correspond to an increase in T_g of 35 – 45K, which is in good agreement to what we measure for MAPLE-deposited glasses.

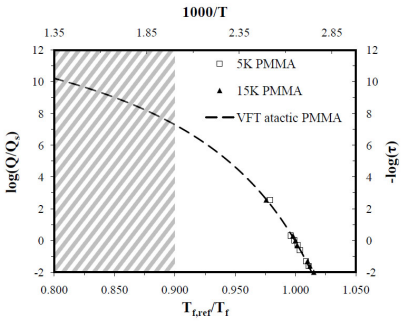

Figure 4: Arrhenius plots of normalized fictive temperature versus normalized cooling rate for 5K and 15K PMMA (left and bottom axes, symbols) and VFT fit for atactic PMMA (right and top axes, dotted line).

Mechanism of MAPLE: Nanostructure. We attribute the unique properties of MAPLE-deposited glasses to the nanostructure. The formation of a nanostructure in polymer films via MAPLE may occur during two distinct stages of the deposition process. In one case, the film may become nanostructured subsequent to material deposition by a mechanism such as evaporation-induced self-assembly, where directional evaporation of the solvent causes 2D ordering in the deposited film. In the second case, structure is set-in prior to the deposition of the desired material atop the substrate. That is, self-assembly of the nanoscale structures occurs within the time frame of the transfer process between the target and substrate. We are concerned with the latter mechanism of nanostructured film formation, as there is no evidence of solvent present in our films. In order to understand the origins of nanostructured films in the MAPLE process, we investigated the structure of sub-monolayer and monolayer films of PMMA deposited atop silicon oxide substrates. These experiments entailed characterizing, both visually and quantitatively, the nanostructure, *i.e.*, nanoglobule morphology, of isolated nanoglobules and monolayer films as a function of deposition time. We also characterized the influence of polymer target concentration on the nanostructure morphology during the early stages of film growth. Our expectation was that by performing

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these measurements, new insight into the mechanism of the nanostructured film formation by MAPLE would be obtained.

To isolate the early-time MAPLE deposition behavior, we investigated the effect of MAPLE deposition time on the nanostructure of the deposited films. Five deposition times were examined (2.5, 5, 10, 20 and 40 min), while maintaining a constant polymer concentration of 0.1% by weight. AFM phase images for three of the resulting depositions (2.5, 10 and 40 min) are shown in Figure 5. The shortest deposition time (see Fig. 5(a)) yielded small, isolated nanoglobules distributed atop the silicon oxide substrate. The substrate was abraded with a razor to confirm the absence of an underlying planar layer. The individual nanoglobules resemble wetted droplets, whose geometry can be approximated as a spherical cap. The globules occupy approximately 16% of the substrate area, and the substrate is visible between the globules. After 10 min of MAPLE deposition, a near-continuous polymer film was formed, consisting of a monolayer of nanoglobules with minimal overlap (see Fig. 5(b)). In this deposition, a small number of globules were deposited on top of other globules, and the substrate was visible only in small discontinuities in the film. This 10 min deposition demonstrated a nanostructured polymer film with an average thickness of just 40 nm. For a deposition time of 40 min, the substrate was covered fully, and more overlapping of nanoglobules was observed (see Fig. 5(c)). To better quantify the differences in film nanostructure, we used image analysis software to measure the size distributions of the nanoglobules. We note that for depositions with times ≥ 5 min, an ultrathin planar film 2-5 nm in thickness was present between the substrate and nanoglobules.

Figure 5d is a histogram plot of the nanoglobule size distribution for the five different MAPLE deposition times. The size distributions for the two shortest deposition times, 2.5 and 5 min, were similar, with a narrow breadth (FWHM = 11 and 13 nm, respectively) and a peak at 20 nm. The three longer deposition times (10, 20 and 40 min) exhibited overlapping, broader size distributions with peaks at 25 nm (FWHM = 22, 23 and 23 nm, respectively). This implies two deposition regimes: an early-time regime, where more of the smaller globules are deposited and a second, slightly broader distribution, which stays constant, even at long deposition times. We suggest two potential mechanisms that may be responsible for the early time deposition regimes. First, the outer surface of the target, which is ablated during very early times, may not be identical in composition to the remainder of the target, due to a thin film of water vapor that freezes on the target prior to loading. Second, the gradual coalescence of deposited globules could be responsible for the initial increase in globule size with deposition time. The existence of two similar deposition regimes was also confirmed for a second target concentration (0.5 wt%). The implication of the dual early-time deposition regimes is that it becomes possible to establish a rule of thumb that the globule size distributions resulting from all depositions longer than the critical time are comparable to each other. This is useful when varying other MAPLE deposition parameters, such as target concentration.

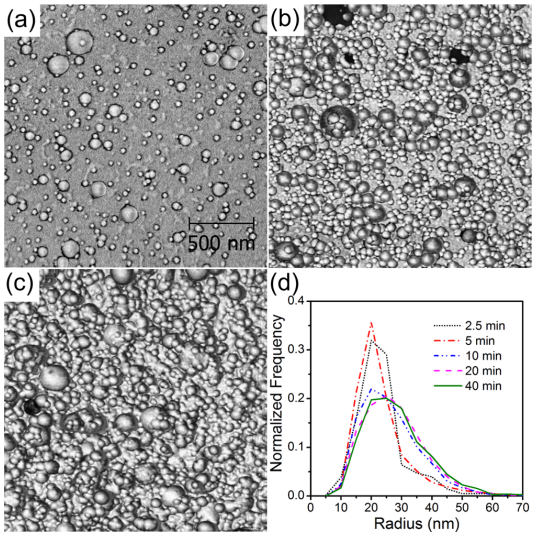

Figure 5. AFM phase images of PMMA films formed after (a) 2.5 min, (b) 10 min, and (c) 40 min of MAPLE deposition time. (d) Histogram plot of nanoglobule radius for five deposition times.

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Earlier work, we briefly addressed the formation of MAPLE-deposited polymer nanoglobules within the context of the Zhigilei model of target ablation in the MAPLE process. Molecular dynamics simulations show that phase explosion causes polymer molecules to be ejected from the MAPLE target within clusters of solvent. In the simulation work, the size distribution of ejected clusters was characterized as a function of fluence and polymer target concentration, where the size was measured in the number of mers of polymer (N = 100 mers per chain) plus molecules of solvent in each cluster. A power law dependence of $N^{-1.6}$ was identified. In Fig. 6(a), the size distribution data for films deposited from 0.05 and 0.1 wt% solutions is plotted with 95% confidence intervals. The best power law fit was found for both 0.05 and 0.1 wt% ($N^{-2.1}$ and $N^{-1.9}$, respectively) over the radius range of 30 nm ≤ R ≤ 65 nm. An intermediate power law fit with exponent of $N^{-2.0}$ was included in the graph in Fig. 6(a). For globules larger than 65 nm, the number of globules present in each film is inadequate for our analysis to generate meaningful statistics, and thus these were not included in the fit. For globules smaller than 30 nm (< $5x10^5$ mers), the data no longer follow a power law expression. An outcome of this study is an explanation for the influence of concentration on the sizes polymer nanoglobules as depicted schematically in Fig. 6(b).

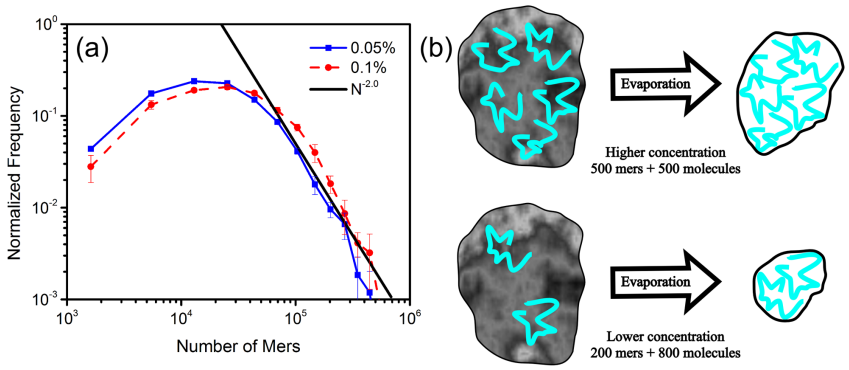

Figure 6: (a) Histogram plot of number of mers per nanoglobule for 0.05% (dotted red curve) and 0.1% (solid blue curve) MAPLE-deposited films, compared with $N^{-2.0}$ scaling. (b) Schematic of nanoglobule formation from MAPLE-ejected polymer/solvent clusters. Equal sized clusters with different polymer concentrations will form differently sized nanoglobules after evaporation.

Properties of Isolated Nanoglobules. In earlier work, (Shepard, APA, 2013) we provided an indirect measurement of nanoglobule stability. In 2013, our focus has been on establishing a protocol to directly measure the thermal stability of isolated nanoglobules prepared via MAPLE. To achieve this, we performed short time MAPLE deposition to generate isolated nanoglobules. We subsequently, used AFM to measure the volume of nanoglobules as a function of temperature. Figure 7 illustrates data from such experiments (ACS Macro Letters 2014). Figure 7a compares the plots of normalized volume (normalized at room temperature) versus temperature for nanodroplets (prepared by annealing nanoparticles) and MAPLE nanoglobules. There is a notable difference in the volume response. Nanoglobules prepared by MAPLE have a pronounced drop in volume at high temperatures while those prepared from nanoparticles do not exhibit such behavior. Figures 7b and c depict profiles of nanoglobules prepared by annealing a nanoparticle and MAPLE, respectively. Cleary, there is a dramatic volume shrinkage at a critical temperature of the MAPLE prepared nanoglobule. I would like to make two important points: 1) The temperature at which the volume reduction occurs is ~ 20 K higher than the bulk glass transition temperature, and 2) The volume reduction is ~ 30% of the film volume. Both of these metrics are consistent with our bulk measurements of MAPLE-deposited films. We hope to expand to nanoscale calorimetry in the future.

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We have build a time of flight measurement system to measure the velocity of isolated polymer nanoglobules during deposition. The time of flight (TOF) measurement system is designed to experimentally measure the time it takes laser-ablated material to travel from the target to the substrate. From this measurement, we can characterize the velocity and kinetic energy of the ablated material, to be used in an energy balance of the material deposition process. Inside the MAPLE chamber, we have installed a 6 MHz quartz crystal microbalance system, positioned directly in front of the ablation plume. The crystal continually oscillates, and the frequency of the oscillation decreases as material is deposited onto the crystal's surface. Using an oscilloscope, we can take a "snapshot" in time of the crystal's oscillatory signal. We can synchronize the beginning of the snapshot to the time when a laser pulse strikes the target, using a photodetector positioned just outside the beam path. The snapshot stored in the oscilloscope is then analyzed using a fast Fourier transform algorithm in MATLAB to pinpoint the time at which the crystal's frequency changed, and hence, what time the material arrived at the crystal sensor. The velocity of MAPLE-deposited material has been investigated only in simulations, where it is reported on the order of 100-1000 m/s. Based on these calculations, we expect the timescale of our measurements will be tens to hundreds of microseconds, which is within the capabilities of the system. I report that the system is working and that we are able to measure velocity of MAPLE-deposited nanoglobules.

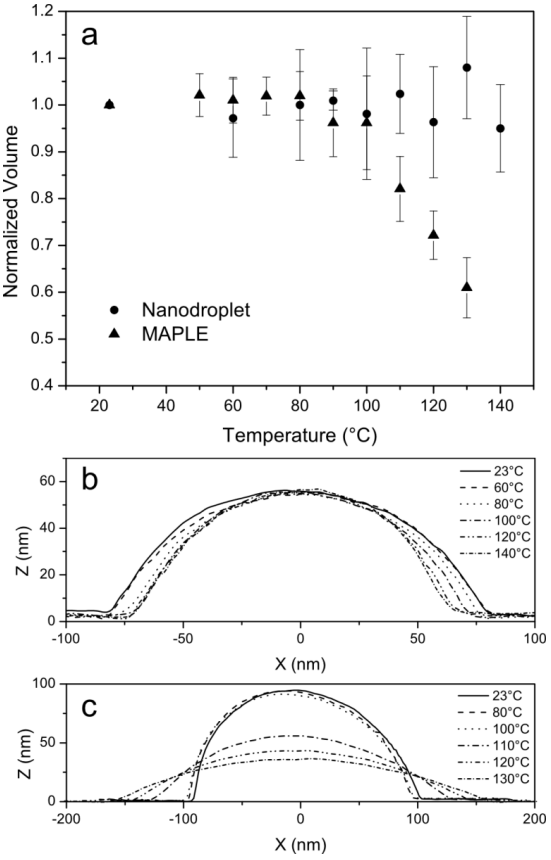

Figure 7: (a) Normalized volume of polymer nanodroplets and MAPLE-deposited nanoglobules as a function of temperature for large features ($V > 10^{-21} m^3$). (b) Profiles of a large nanodroplet's XZ cross-section as it is heated. (c) Profiles of a large MAPLE-deposited nanoglobule's XZ cross-section as it is heated.

Macromolecular Deposition of Semi-Crystalline Polymers via MAPLE. We have very recently begun the gas-phase deposition of semi-crystalline polymers via MAPLE. Figure 5 displays Atomic Force Microscopy (AFM) images of a model crystallizable polymer, polyethylene oxide (PEO) deposited *via* MAPLE onto native silicon substrates held at room temperature. Panels A, B, and C illustrate height images of a film deposited at ~ 0.0001 nm/sec for 120, 240, and 480 min, respectively. While panel D is the phase image of panel C. The images demonstrate that the size of the monolamellae increases with increasing deposition time. This phenomenon is a result of the films growth mechanism *via* the gas phase deposition process and illustrates a new means for engineering the morphology in semi-crystalline polymer thin films similar to that of growing crystalline films of small molecules via vapor deposition.

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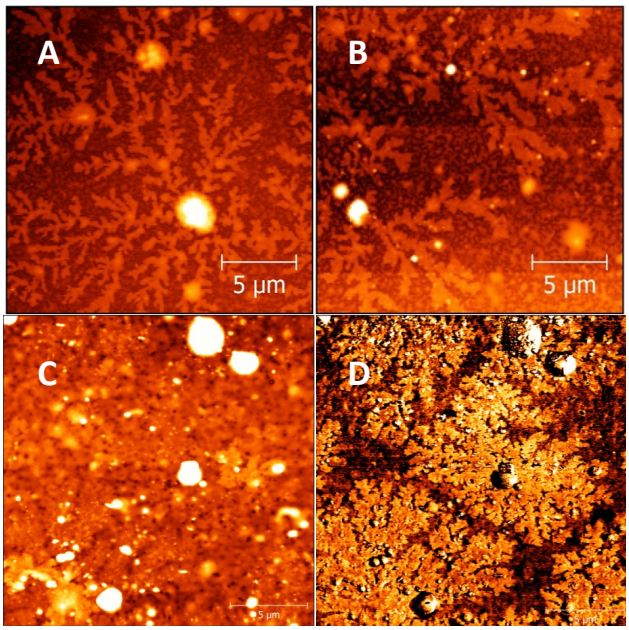

Figure 5: Development of dendritic arms of PEO on Si wafers with surface coverage after (A) 120 min (B) 240 min (C)-(D) 480 min deposition times. Panels A-C are height images while panel D is a phase image.

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Abstract

In 2011, we discovered that polymer films with exceptional thermal and kinetic stability could be formed by Matrix Assisted Pulsed Laser Evaporation (MAPLE) onto substrates held at low temperatures compared to the glass transition temperature (Tg) of the polymer. The unique and unprecedented combination of properties is due to the film morphology, i.e., the films are nanostructured. The aim of this proposal was to understand the mechanism of film formation and to characterize the nanoscale building blocks of the stable glasses. Recently, we characterized the transport, i.e., time-of-flight, and nanoscale thermal properties of amorphous polymer nanoglobules fabricated via Matrix-Assisted Pulsed Laser Deposition (MAPLE). We reported the first experimental measurement of the velocity of polymer during MAPLE processing and its connection to nanostructured film formation. A nanoscale dilatometry technique using atomic force microscopy was employed to directly measure the thermal properties of MAPLE-deposited polymer nanoglobules. Similarly to bulk stable polymer glasses deposited by MAPLE, polymer nanoglobules were found to exhibit enhanced thermal stability and low density despite containing only thousands of molecules. By directly connecting the exceptional properties of the nanostructured building blocks to

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those of bulk stable glasses, we gained insight into the physics of glassy polymeric materials formed via vapor-assisted techniques.

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Archival Publications (published) during reporting period:

K. Shepard, D. Christie, C.L. Sosa, C.B. Arnold, R.D. Priestley*, Patchy Janus Particles with Tunable Roughness and Composition via Vapor-Assisted Deposition of Macromolecules, submitted.

K.B. Shepard, C.B. Arnold, R.D. Priestley*, Transport and Stability of Laser-Deposited Amorphous Polymer Nanoglobules, ACS Macro Letters, 3, 1046 (2014)

K. Shepard, C.B. Arnold, R.D. Priestley*, Origins of Nanostructure in Amorphous Polymer Coatings via Matrix Assisted Pulsed Laser Evaporation, Applied Physics Letters, 103, 123105 (2013)

K. Shepard, R.D. Priestley*, MAPLE Deposition of Macromolecules, Macromolecular Chemistry and Physics, 214, 862 (2013)

K. Shepard, Y. Guo, C.B. Arnold, R.D. Priestley*, Nanostructured Morphology of Polymer Films Prepared by Matrix Assisted Pulsed Laser Evaporation, Applied Physics A, 110, 771

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Reporting Period

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Research Objectives

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